Message

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**Sent:** 5/3/2018 6:47:50 PM
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**Subject:** Update on serum method
**Attachments:** 05032018 orbitrap calibration curves.pptx; ExcelExp_Long_180503112205.xls

Hi all,
Here's an update on the serum method development.

1. PFAS contamination:
James and Mark manually purged the LC system using DI and methanol and the legacy PFAS contamination went away. After running a few standards, though, the compounds started showing up again in the blanks. However, the area counts are lower than before and PFHxS and PFOS standard curves look much better (see attached powerpoint). We may have to accept some background on this instrument.

Contamination increases slightly over the course of the run (see blanks tab on the data spreadsheet). We could include regular blanks (e.g., every 5 samples) and track background for every run. Whether a sample contains a compound or not would be dependent on the background levels for that run.

With the May 3rd run, we saw some carryover of GenX in the blanks. Area counts were relatively low but still above one of the 0.5 ng/mL standard injections. We have not seen significant carryover of Nafion bp1, Nafion bp2, PFO4DA, PFO5DoDA, or the GenX internal standard.

2. We reran the standards and serum extracts from last week.
GenX standard curves from the two runs look similar. Relatively good linear fit between 0.5 and 20 ng/mL
None of the serum samples had detectable GenX except one that had a clear peak, although this is suspect since we didn't see this in the run from 4/25. All samples will be rerun when we're out of the method development phase.
Nafion byproduct 1 response is still poor below 10 ng/mL.
Nafion byproduct 2 concentrations are between 1 and 7 ng/mL for the 10 serum samples.
We cannot measure for PFMOAA

3. SRM1957 results:

| | Reference values | Apr-25-2018 run | May-03-2018 run |
|---|---|---|---|
| PFOS | 21.1 +/- 1.2 | 23.7 | 15.8 |
| PFOA | 5.00 +/- 0.40 | 4.2 | 3.7 |
| PFNA | 0.88 +/- 0.07 | ND | 0.9 |
| PFDA | 0.39 +/- 0.10 | 1.7 | 0.2 |
| PFHxS | 4.00 +/- 0.75 | 6.5 | 3.2 |

We see better measurement of PFNA and PFDA in SRM1957 on the May 3rd run, possibly from the cleaner baseline. The standard curves for the legacy compounds have not been corrected with IS yet. Our measurements of the SRM1957 will be more accurate with IS correction.

4. Next steps

Update our standard curve to include all compounds from Chemours: PFO2HxA, PFO3OA, PFO4DA, Nafion byproduct 4, NVHOS, PMPA, PEPA, PFO5DoDA

Start using the full suite of internal standards for the legacy compounds so we can do IS correction.

Should we normalize Nafion byproduct 2 standards with any of the mass labeled compounds we have (i.e., mass labeled GenX or the legacy PFAS)?

Prepare calf serum QCs at 2 and 15 ng/mL to include in every run. Ask Chuhui if I can use her preparation of the emerging PFAS for these QCs.

Data are attached. Thanks,
Nadine